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HEAT STERILIZABLE, IMPACT RESISTANT CELL DEVELOPMENT

JET PROPULSION LABORATORY
CONTRACT NO. 951296

REPORT FOR SECOND QUARTER
1967
APRIL 1 TO JUNE 30, 1967

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JPL CONTRACT 951296

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ABSTRACT

Previous reports on this contract were the Annual Report covering the period from September 1965 to December 1966 and the Report for the First Quarter of 1967. Work during the Second Quarter may be summarized as follows.

Six of the original twenty sealed silver-zinc cells made with sterilized components still remain on test after 62 deep discharge cycles, and a similar cell constructed to study gassing with constant current charging has failed after 77 deep cycles. The pressure was 15.5 psig at failure.

So far, 18 cells have been constructed in PPO cases, sealed, sterilized, and cycled through the required four cycles and placed on stand or float. One cell on stand and one on float have been discharged after the first month. There was no loss of capacity in the one on float, whereas there was a considerable loss on stand. Additional work was done with other cells on high temperature stand. Cells standing one month at 160°F, which might be equivalent to a room temperature stand of 16 to 20 months, showed a capacity loss of about 40%. Capacities on recharge were good.

Gas generation is being studied in sealed cells. So far pack tightness, separator type, position of separator, type of additive and the couple effect between zinc and its silver grid have been investigated. Gas generation in some measure continues to be unpredictable.

The study of sealed, sterilized silver-cadmium cells on stand, cycle, or float was in part set back by a leaking sterilization bomb which affected one entire group of test cells. The work is again underway. Emphasis is at present on the performance of SWRI-GX separator in this series of tests rather than on RAI-116 separator.

Highly reliable case-to-cover seals have been obtained with PPO 531-801 using an epoxy adhesive. However, a crack-free cover of this material,

with silver terminals molded in, has not been achieved. On the other hand, covers of PPO 541-801, containing molded-in silver terminals, have been free of cracks, but satisfactory case-to-cover seals with epoxy adhesive have not been achieved with this plastic.

Hot gas welded seals on PPO 541-801 have not been attainable.

Shock testing of silver-zinc cells in which the electrodes were reinforced with plastic struts produced severe mechanical failures at the 10,000 g impact. The ensuing design is with sheet metal reinforcement of the electrodes, and these cells are currently being fabricated for shock testing.

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INTRODUCTION

The first published report on Contract 951296 was the Annual Report covering the period September 24, 1965 through December 31, 1967. That report outlined the requirements for a high energy density cell - either silver-zinc or silver-cadmium - which could be heat sterilized in the sealed but unformed condition, formed, impacted as in a hard planetary landing, and function thereafter through four discharge-charge cycles. The division of work between ESB-Research Center, ESB-Exide Missile and Electronics Division, and the Jet Propulsion Laboratories was described. Progress during the stated period toward the above-mentioned goals was reviewed.

A report covering the First Quarter of 1967 was issued early in May. This report brought the work up to date, confirming some of the earlier conclusions about optimum components and designs, and revising others.

The present report, for the Second Quarter of 1967, continues the study and emphasizes the observation of excessive pressure encountered during formation of sterilized, sealed silver oxide-zinc cells in certain instances. The inadequacy of earlier cell structures to resist the very high shock tests is discussed, and the plans for coping with this problem are outlined.

I. PERFORMANCE OF SEALED CELLS

A. Cells with Sterilized Components

Six of the original twenty sealed AgO-Zn cells made with sterilized components remain on test after 62 cycles. Cell #17 failed after the 52nd cycle and cell #20 after the 57th cycle, both apparently due to silver and zinc penetration through the separator. Cycling was interrupted after the 53rd cycle and the cells stood on open circuit for 17 days while the automatic cycling equipment was used for other tests. Cycles 54 through 62 were then run on another cycling apparatus; however, it was discovered after the 62nd cycle that a malfunction in this cycler was causing cutoff on charge as low as 1.92 v instead of the prescribed 1.96 v. This resulted in low apparent capacities for some cells. Table I lists a summary of discharge capacities obtained during this quarter.

The sealed cell constructed for the study of gassing with constant current cycling has failed due to shorting after 77 cycles. Pressure in the cell at failure was 15.5 psig. Gas chromatographic analysis indicated the presence of hydrogen, but practically no oxygen. Penetration of the RAI-110 separator by both silver and zinc was observed, particularly at the first layer next to each respective plate. Significant loss of negative active material had occurred, especially at the top and sides of the plates.

Table II summarizes cell discharge data for cycles 50-75. Note that total capacity could be increased by using stepwise charge and discharge schemes as in cycle #50.

B. Cells Sealed in PPO Cases Prior to Sterilization

The group of six cells sterilized in sealed PPO cases have completed four electrical cycles; three are now on float at 1.96 v and three on open circuit stand. These cells will be discharged periodically to determine capacity, then recharged and returned to float or stand.

Twelve additional 5-plate AgO-Zn cells sealed in PPO cases were constructed, sterilized, and cycled in the same manner as the previous six, except for the use of new rather than used cases, and PPO rather than Teflon shims. All passed the sterilization procedure without leaking.

Cycle data for the 18 cells, plus those for two additional cells identical in construction and cycling but not sterilized, are given in Table III. The first set of sterilized cells are numbered 1 to 6, the second group are 1A to 6A and 7 to 12, and the non-sterilized controls are C-1 and C-2. Sterilization apparently causes capacity loss and probably contributes to scatter of the data. The controls show excellent agreement throughout cycling.

TABLE I

Discharge Capacities of Twenty Cells
Sealed After Sterilization

Cell #	Description	Discharge Capacity (amp-hr) Cycle				
		40	45	50	55‡	60‡
11	RAI-110 (125°C)	3.6	3.4	3.3	-	-
13	"	3.6	3.6	3.6	-	-
14	"	3.4	3.4	3.5	-	-
15	"	3.5	3.3	3.5	-	-
17	RAI-110 (135°C)	2.8	2.4	3.5	*	-
18	"	2.8	2.3	2.6	-	-
19	"	2.6	2.5	2.6	-	-
20	"	3.3	3.1	3.3	-	**

‡ Capacity data for cycles 54 through 62 are omitted, due to apparatus malfunction which caused cells to cutoff prematurely on charge.

* Cell #17 failed after 52 cycles.

** Cell #20 failed after 56 cycles.

TABLE II
Gassing of Sealed AgO-Zn Cell on
Constant Current Charge

Cycle #	Charge Current (ma)	Input (amp-hr)	Discharge Capacity (amp-hr)	Pressure (psig)
50	100, then 35	2.75 +0.23	2.91 at 1.0 A +0.43 at 0.5 A	14.5
55	150	2.51	2.47	14.0
60	150	2.61	2.57	14.5
65	150	2.28	2.16	15.5
70	150	2.23	2.15	15.5
75	150	2.69	2.15	16.0

Cell failed after 77 cycles.

TABLE III

Cycle Tests - Cells Sealed before Sterilization

Cell #	Discharge Capacity (amp-hr) Cycle				Preparation for Float or Stand	
	1	2	3	4	Input (amp-hr)	State
	(a)	(b)	(b)	(b)	(b)	
1	3.0	2.4	2.2	2.5	2.6	F
2	3.2	2.5	2.4	2.5	2.6	S
3	3.0	1.9	1.8	2.0	2.2	F
4	2.9	2.7	2.6	2.7	2.7	S
5	3.5	3.6	3.6	3.3	3.2	F
6	3.2	3.0	2.8	2.9	2.8	S
	(a)	(c)	(c)	(b)	(b)	
1A	2.3	2.4	2.7	2.5	2.6	F
2A	2.3	2.5	2.9	2.9	3.0	S
3A	2.7	2.9	3.0	2.7	2.9	F
4A	1.8	2.1	2.4	2.7	2.8	S
5A	2.0	2.2	2.4	2.6	2.7	F
6A	2.9	2.9	3.0	2.8	2.9	S
	(a)	(b)	(b)	(b)	(b)	
7	4.2	3.5	3.5	3.1	3.0	F
8	3.2	2.6	2.5	2.5	2.5	S
9	1.7	1.6	2.2	2.2	2.4	F
10	3.8	3.2	3.1	2.7	2.7	S
11	2.7	2.3	2.7	2.5	2.6	F
12	2.8	2.2	2.0	1.8	1.9	S
	(a)	(b)	(b)	(b)	(b)	
C-1	3.5	3.8	4.1	3.6	3.4	F
C-2	3.6	3.7	4.4	3.9	3.5	S

(a) 70 ma charge to 1.96 v.

(b) 100 ma " " " .

(c) Two-step charge: 100 ma to 1.96 v, then 35 ma to 1.96 v.

All discharges were at 1.0 A to 1.2 v.

Two of the first set of sealed PPO cells, one on stand (S) and the other on float (F), were discharged after one month's time and again after the second month. Capacities were as follows:

Cell #	State	Month	Input Prior to Float or Stand	Discharge Capacity
5	F	1st	3.22 amp-hr	3.31 amp-hr
		2nd	3.04	3.52
6	S	1st	2.79	2.20
		2nd	2.76	2.62

C. Cells on High Temperature Stand

Some of the earlier cells that had been constructed to study various electrochemical properties of the system after sterilization were placed on stand at the elevated temperature of 160°F. This temperature was selected because it would accelerate any capacity robbing reaction and give some indication of what type of stand life on sterilized cells could be expected at ambient temperatures. If it is assumed that increasing the temperature by 18°F doubles the rate of the reaction, a stand period of one month at a temperature of 160°F would be expected to produce a loss similar to that of approximately 16 to 20 months at room temperature. This has not been verified for temperatures of this magnitude, however. The results of this study are given in Table IV. Generally results indicated a capacity loss of 13-18% during the first 15 days; and 37-40% during the first 30 days. After 45 days two of the four cells had either shorted or had nearly done so, so that shorting occurred during the subsequent recharge. Cells that had been on stand up to 30 days at 160°F had not been damaged, and after recharging gave nearly as much capacity in some cases and more capacity in others than they did before the start of stand.

II. EFFECT OF EPOXY RESIN ON CELL PERFORMANCE

Test A. The cells made with components which had been sterilized in the presence of PPO material and epoxy sealant, as described in the previous Quarterly Report, were run for two additional cycles. By this time the two cells apparently damaged by epoxy had almost completely recovered to normal capacity.

Test B. Six more 3-plate Ag-ZnO cells were constructed, and two of these were sterilized in the presence of each of the following:

TABLE IV
Capacity Retention After Stand at
160°F

Cell No.	22	25	26	27	28	34	37	38	39	40	44	45	48
Separation	(1)	(1)	(1)	(1)	(1)	(2)	(3)	(2)	(4)	(1)	(1)	(1)	(2)
Capacity in AH before Stand	4.6	4.4	4.6	5.6	4.6	4.5	4.5	5.0	5.0	5.1	4.6	4.8	4.6
Days on Stand	45	45	45	45	70	30	30	30	30	30	15	15	15
Temperature	160°F	160°F	160°F	160°F	RT	160°F	160°F	160°F	160°F	160°F	160°F	160°F	160°F
OCV After Stand	1.57	1.54	1.60	1.60	1.86	1.81	1.84	1.58	1.58	1.57	1.85	1.84	1.84
Capacity in AH After Stand	0.5	shorted	0.7	0.8	4.2	2.4	2.4	3.0	3.2	1.6	4.2	3.8	3.8
Per cent of capacity retained	11	0	15	14	91	53	53	60	60	31	87	82	82
Capacity in AH following recharge after discharge following stand	shorted	shorted	1.2	1.3	-	5.0	4.4	4.5	6.1	5.6	5.5	4.9	5.2

NOTES: (1) Separator code: (1) 5 layers RAI-116
(2) 5 layers RAI-110
(3) 5 layers SWRI-GX
(4) 5 layers SWRI-G

(2) Cell 27 contained ZnO-HgO negatives and was unsterilized; all others contained ZnO-Compound 323-43 and were sterilized.

1. Epoxy DEN438-EK85 with DMP-30 catalyst coated and cured onto a PPO 531-801 shim.

2. PPO 531-801 shim.

3. Control cells not in contact with either PPO material or epoxy.

These cells were sterilized at 135°C for 136 hours. After sterilization the cell packs and shims were placed in open Plexiglas cases. The cycle data, reported in Table V, indicates no damage due to epoxy occurred in this test.

Test C. A test was run to observe the effect of epoxy in the ground condition on cell capacity. The following cells were constructed in duplicate:

1. With epoxy DEN 438-EK85 + 5 wt. % DMP 30 cured 16 hr. at 25°C, 2 hr. at 50°C, 2 hr. at 75°C and 16 hr. at 100°C (Experiment 76).

   (a) Sterilized epoxy liquor in a sterilized cell.

   (b) " " " " an unsterilized cell.

   (c) Epoxy chips in a sterilized cell.

   (d) " " " an unsterilized cell.

2. The same experimental scheme was followed using the same epoxy and catalyst with 10 wt. % DER741A and the same curing schedule (Experiment 78).

3. Control - 3 plate Ag-ZnO cell unsterilized. No contact with epoxy.

The sterilized epoxy liquor was prepared by placing 1.5 gms of ground up epoxy in 50 mls of 40% KOH in a Teflon container and sterilizing in a nickel bomb at 135°C for 120 hrs. Upon opening the bombs after sterilization a distinct MEK odor was noticed, but this disappeared after the electrolyte sat overnight in a closed polyethylene container.

The cells were constructed with wrapped negatives using RAI-110. Cycling of these cells was carried out in open cell cases. Charge rate was 35 ma for formation and 50 ma on subsequent recharges. Discharge was at 600 ma to 1.20 v cutoff. Cycle data is given in Table VI.

It appears that the epoxy may have caused some damage on the first cycle, but, in all cases except one, the cells have recovered on the second and subsequent cycles. The "78-Liquor-Sterilized" (Experiment 78) cells were

TABLE V
Effect of PPO + Epoxy on Cell Performance
Test B

Cycle	Cell #	Additive	Chg.	Dischg.	Mid Volts
1	1	Epoxy	2.36	2.27	1.33
(35 ma)	2	"	2.18	2.12	1.33
	3	531-801	2.26	2.13	1.32
	4	"	2.29	2.21	1.34
	5	Control	2.31	2.07	1.31
	6	"	2.35	2.16	1.32
2	1	Epoxy	2.27	2.04	1.36
(70 ma)	2	"	2.15	2.00	1.36
	3	531-801	2.11	1.96	1.32
	4	"	2.14	2.05	1.37
	5	Control	2.18	1.78	1.30
	6	"	2.27	2.02	1.35
3	1	Epoxy	2.34	2.03	1.36
(70 ma)	2	"	2.27	1.90	1.34
	3	PPO-531	2.73	1.85	1.33
	4	"	2.73	1.99	1.37
	5	Control	2.27	1.87	1.33
	6	"	2.17	1.94	1.32
4	1	Epoxy	2.24	2.10	1.38
(70 ma)	2	"	2.14	1.97	1.36
	3	PPO-531	2.24	1.98	1.36
	4	"	2.24	1.99	1.37
	5	Control	2.24	1.93	1.37
	6	"	2.10	1.94	1.37

TABLE VI
Effect of Epoxy on Cell Performance
Test C

Cell No.	Description *	Cycle #1 Cap	Cycle #1 Mid Volts	Cycle #2 Cap	Cycle #2 Mid Volts	Cycle #3 Cap	Cycle #3 Mid Volts	Cycle #4 Cap	Cycle #4 Mid Volts
1	76-Liq-Ster	1.92	1.42	2.47	1.41	2.37	1.42	2.28	1.44
2	" " "	2.19	1.41	2.37	1.41	2.23	1.41	2.23	1.44
3	76-Chip-Ster	1.56	1.41	2.43	1.39	2.22	1.41	2.09	1.42
4	" " "	1.57	1.41	2.49	1.39	2.44	1.42	2.42	1.44
5	78-Liq-Ster	1.38	1.36	1.93	1.38	2.01	1.38	1.95	1.42
6	" " "	1.38	1.35	1.44	1.32	1.60	1.33	1.62	1.39
7	78-Chip-Ster	1.39	1.38	2.27	1.38	2.23	1.41	2.10	1.44
8	" " "	1.58	1.38	2.13	1.38	2.11	1.40	2.04	1.43
9	76-Liq-Unster	1.86	1.42	2.55	1.32	2.49	1.38	2.36	1.45
10	" " "	1.70	1.42	2.51	1.30	2.44	1.36	2.35	1.44
11	76-Chip-Unster	1.59	1.41	2.52	1.40	2.37	1.41	2.14	1.45
12	" " "	2.03	1.41	2.18	1.38	2.13	1.40	2.07	1.44
13	78-Liq-Unster	1.71	1.41	2.40	1.28	2.41	1.36	2.10	1.45
14	" " "	2.12	1.41	2.21	1.30	2.12	1.36	2.04	1.44
15	78-Chip-Unster	1.86	1.41	2.57	1.40	2.24	1.42	2.04	1.45
16	" " "	2.31	1.39	2.30	1.38	2.14	1.38	1.98	1.43
17	Control Unster	1.91	1.40	2.13	1.41	2.08	1.41	1.95	1.44
18	" "	2.04	1.40	2.34	1.30	2.27	1.32	2.22	1.43

* 76 and 78 refer to Experiments 76 and 78.

most severely damaged on the first cycle and have yielded the poorest capacities on the remaining cycles. These cells, however, have shown some gradual improvement with cycling.

III. GAS GENERATION IN SEALED CELLS

Considerable data generated during the last reporting period indicated higher cell pressures were being generated in sealed AgO-Zn cells having in them features permitting heat sterilization than was true of normal cells, particularly during the formation charge. Wide variation in the amounts of these pressures was evident, these seemingly being associated with the separation used in construction. Cells having chemically cross-linked RAI-116 generally developed greater pressures than those having radiation cross-linked RAI-110. There was, however, an overlapping of data which suggested that other factors were probably involved. Further study of some of these variables was made during this period.

A. Cell Pack Tightness

One variable studied was that of cell pack tightness. One of the characteristics of grafted separators is that expansion occurs during equilibration with electrolyte. The amount of expansion is related to the concentration of electrolyte, being greater in dilute electrolyte than in concentrated, and to the temperature during equilibration. Examination of the cell components after sterilization indicated that this expansion would occur, regardless of the expansion space provided, at the expense of other cell components. In this instance the zinc oxide electrode was compacted, it being the most easily compressed element in the cell. Since this variable could be more easily studied in cell stacks being placed in actual cell jars and since sterilizable cell jars were not available at the time, a preliminary study was made using unsterilized cells. These were seven-electrode cells having plate dimensions of 1 3/4 in. by 1 3/4 in. Separation consisted of 5 layers of grafted polyethylene, chemically cross linked, produced by Southwest Research Institute. The dry thickness of this material is approximately 0.0013 in. per layer. Proper spacing was selected to allow expansion to 0.0014, 0.0017, and 0.0021 in. per layer in three cells. Data on these cells are found in Table VII. The data suggest that the internal pressure tended to be highest in the more tightly packed cell and lowest in the more loosely packed one. Discharge voltage and capacity tended to diminish as separator expansion allowance increased.

Data obtained recently on cells with sterilized cell packs are not as conclusive as those using unsterilized cells.

B. Cells with Positives Wrapped

Six cells (5-plate Ag-ZnO) have been constructed by wrapping the positive plates with separator to test the effect of this assembly on the amount of

TABLE VII

Internal Pressure as a Function of Cell Pack Tightness

Cell No.	68	69	70
Separator thickness allowance per layer	0.0014	0.0017	0.0021
Formation charge acceptance at 6 ma per sq. in; AH per gm Ag	0.37	0.37	0.36
Pressure change during formation, in.Hg g	63	13.6	13.0
First discharge capacity; AH per gm Ag at 100 ma per sq. in.	0.28	0.28	0.26
Median voltage	1.43	1.40	1.38
Recharge mode, CP 1.96 v for X hrs	90	90	73
final current; ma	< 1	< 1	1
final pressure; in.Hg g	83.2	20	15
Pressure after 72 hrs. OC; in.Hg	79.2	13.6	9.8
Second discharge capacity; AH at 100 ma per sq. in.	5.85	5.22	5.50
continued at 20 ma per sq. in.	0.75	0.59	0.68
Total capacity; AH	6.60	5.81	6.18
Capacity, AH per gm Ag	0.36	0.32	0.34

gas produced. It is well known that changes in electrolyte levels and concentrations occur on charge and discharge in alkaline cells where membranes are present. Thus, the magnitude of these changes should depend on the volume enclosed within the separator placed around the electrode. By wrapping the positives the major volume of the cell is now given over to the zinc electrode and concentration changes should be minimized for whatever effect this might have on gas production from the negative plate. Since all of the twenty cells and the PPO cells were negative wrapped, some data on positive wrapped cells was also desired. Three cells were made using RAI-116 and three using RAI-110. They were then sterilized at 135°C for 120 hrs. The cell packs were sealed into polystyrene cases, fitted with pressure gauges and overpotted with epoxy.

One of the RAI-116 wrapped cells developed excessively high pressure on formation and was removed before the cycling tests. When the cell was removed from the charger, pressure was at 120 psig. The cell maintained a 1.87 v open circuit voltage, but within 3 hours the pressure had risen to 129 psig. The cell was disassembled and a sample of the gas collected. Analysis showed only traces of hydrogen present; however, since an unknown quantity of air was inadvertently mixed with the gas sample, the actual level of hydrogen in the cell was not determined. Neither the positive nor the negative plates were completely charged. Some Zn dendrite growth was noticed at the bottom of the cell case which may have contributed to the gassing. Dendrite growth appears to be much greater in these positive-wrapped cells; however, excessive pressures were not attained during formation of the remaining five cells.

The remaining cells completed four cycles and are now on float or stand test. Cycle and pressure data are given in Table VIII. It appears that the cells have reached equilibrium pressures by the fourth cycle. Table IX gives pressure data for previously tested negative wrapped cells for comparison. No real difference in pressure generation between positive and negative wrapped cells is noted.

C. The Couple Effect

It was suggested that a large part of the gas build-up during the formation of a Ag-ZnO cell might be due to the couple formed between the zinc negative and its silver grid as demonstrated by Snyder and Lander*. Usually HgO is added to the zinc mix and the mercury produced during formation gives protection against the phenomenon. However, with the present cells in which

* R. N. Snyder and J. J. Lander, Rate of Hydrogen Evolution of Zinc Electrodes in Alkaline Solutions, Electrochemical Technology, 3, 161 (1965).

TABLE VIII

Cycle and Pressure Data: Positive Wrapped Cells

Cell #	Discharge Capacity (amp-hr) Cycle 1	2	3	4	Preparation for Float or Stand Input (amp-hr)	State
1-110	3.6	3.2	3.6	3.3	3.6	S
2-110	2.8	3.5	3.3	3.5	3.2	F
3-110	3.4	3.9	2.6	3.4	3.4	F
4-116	3.8	3.3	3.3	3.1	3.0	F
5-116	3.1	2.9	3.0	2.8	2.9	S
	Pressure (psig)					
1-110	7	9	9	9	9	
2-110	0	3	3	5	5	
3-110	4	6	7	8	8	
4-116	10	15	15	17	17	
5-116	3	5	7	7	7	

Formation was at 70 ma; subsequent cycles, 100 ma charge.

Discharge was at 1.0 A for all cycles.

TABLE IX

Pressure Data for Negative Wrapped Cells *

Cell #	Separator	Sterilization Temp.	Pressure (psig) Cycle 1/7	5/8
5	RAI-116	135°C	7	8
8	"	125	12	16
11	RAI-110	135	< 4	< 4
16	"	125	5	5
CC	"	135	6	11

* Data from Pages 6 and 7 of the Report for First Quarter Jan. 1 to March 31, 1967. Cell CC was charged at constant current whereas the others were charged at constant potential.

Compound 323-43 is used, it was proposed that the zinc might charge before the additive becomes effective, and this might lead to the observed gas pressure. To test this hypothesis and evaluate grid materials a number of experiments were carried out using regular gassing tubes. Various pieces of other metals were fastened to zinc sheets 1 inch by 1 inch square, and these combinations were placed in gassing tubes in 40% KOH. The data are shown in Table X. Amalgamations, when used, were carried out in 0.3% $HgCl_2$ solution or electrolytically from a solution of potassium mercuric iodide.

The data indicate that using bare Ag as a grid in the presence of zinc would produce about 8 times as much gas as when the Ag grid was amalgamated. Moreover, amalgamated zinc in the presence of pure silver produces very little gas. As might be expected, the best results were obtained when both the zinc and the silver were amalgamated. For the metals tested, the order of protection appears to be Cu > Pb ≈ In > beryllium copper > Cd ≈ Ag > Ni.

After having studied the couple effect of dissimilar metals with sheet zinc in gassing tubes, the experiments were extended to ZnO powder electrodes gassing on formation. To do this a special cell was made from a polyethylene bottle the diameter of which was slightly larger than the width of the ZnO electrode and the height greater than that of the electrode. The bottom of the bottle and two U-shaped portions of the side wall, except for two narrow reinforcing bands, were cut away. The top of the bottle was sealed to an inverted buret, and a ZnO electrode, enclosed in its retainer (EM 476), two sheets of perforated, corrugated PVC, and fibrous sausage casing wrapping, was inserted as a tight diameter in the bottle. The bottle was placed in a polyethylene beaker over U-fold of fibrous sausage casing containing silver electrodes. The bottle was oriented so that the windows cut in the side wall were opposite the positive electrodes. Separators were used to direct the flow of any hydrogen or oxygen gas.

As shown in Table XI an electrode made with ZnO and Compound 323-43 produced no gas when charged in 43% KOH nearly saturated with ZnO to 55% of theoretical capacity, and produced only 0.4 ml of gas when left on open circuit in 40% KOH for 65 hours. On the other hand, an electrode without Compound 323-43 produced 4.1 ml of gas while charging to 47% of theoretical capacity and produced 17 ml of gas on open circuit stand for 65 hours. When a 5% amalgamated silver grid and an electrode mix in which Compound 323-43 was left out is used, only 1.4 ml of gas is produced by the end of charge to 47% of theoretical capacity. The volume of gas produced after 65 hours stand on open circuit was also low, namely 1.5 ml.

These results suggest several conclusions. The first of which is that in loosely packed cells Compound 323-43 is effective in giving gassing protection both on charge and open circuit stand. The second is that gassing reduction on charge can be reduced significantly if the grid be amalgamated. Thus, the so-called couple effect does operate but can be minimized.

TABLE X

Volume of Gas (ml) from Couples in 40% KOH at a Given Time

Electrode	5 hrs.			24 hrs.			48 hrs.			168 hrs.			400 hrs.	
	1*	2*	3*	1	2	3	1	2	3	1	2	3	1	2
Zn + Ag wire	0.3	0.3	0.4	2.7	4.0	3.3	2.7	4.0	3.3	3.5	5.3	4.2		
Zn + Cd	0.8	0.6		2.9	2.6		3.4	3.4						
Zn + Ni	5.6	3.0		8.5	4.3		9.5	4.6						
Zn + Be - Cu	1.0	1.4		1.7	2.3		2.0	2.6						
Zn + In	0.3	0.5		1.0	1.7		1.4	2.3						
Zn + Pb	0	0		1.8	1.4		2.5	1.8						
Zn + Cu	-			-			0.6	0.7		0.5	0.7		1.1	1.8
Zn-Hg+Ag-Hg	-			-			0	0		0	0		0	0
Zn + Ag - Hg	-			-			0.4	-		0.6	-		0.8	-
Zn-Hg + Ag	-			-			0	0.2		0.1	0.3		0.3	0.5

* Replicates

TABLE XI

Volume of Gas as a Function of Electrode Conditions in 40% KOH

Electrode	Current (mA)	Time (hr)		Vol. of Gas (ml)	
		Charge	Stand	Charge	Stand
ZnO-Compound 323-43, Ag Grid	100	1		0	
	100	18		0	
	0		10		0.0
	0		65		0.4
ZnO, Ag Grid	35	2		0.1	
	35	40		3.2	
	35	48		4.1	
	0		20		4.0
	0		50		12.0
	0		65		17.0
ZnO, 5% Amal. Ag Grid	21	6.4		0	
	21	73		1.4	
	0		20		0.0
			30		0.9
			65		1.5

* Charged in 43% KOH nearly saturated with ZnO

IV. PERFORMANCE OF SEALED SILVER-CADMIUM CELLS WITH SWRI-GX UNDER VARIOUS CYCLING REGIMES

With the receipt of approved separator material and the demonstration of satisfactory cycling performance of the test vehicle using this material, sterilization of the required cell stacks was completed. Forty-eight assembled stacks were placed in nickel containers - eight stacks per container, three containers per bomb. Polysulfone plates were inserted at each end of the can to fill it out and keep the cell stacks upright. The cans were filled to above the plate tops with 40% KOH, inserted into stainless steel bombs, and sterilized at 135°C for 120 hours in a circulating air oven. Leakage of the bombs resulted in some water loss from the electrolyte so that approximately the upper third of each cell stack was not immersed in electrolyte. Electrolyte concentration increased from 40% to 48% KOH. The latter value is based on a composite sample taken from each of the six cannisters so that a higher concentration could have resulted in individual cans.

Two of the cell stacks were built into cells (Table XII) which were in the flooded condition and open and were then discharged to provide an indication of the effects of the bomb leakage. Both charged normally and yielded 1.58 and 1.84 ampere hours on discharge within the expected range.

The remaining cell stacks were then similarly assembled, formed and discharged. Five cells showed poor charge acceptance on formation and poor discharge capacities. The average capacity of the remaining cells was 1.48 $\pm$ 0.27 amp-hr to an 0.80 volt cut-off. The range was from 1.00 to 1.77 amp-hr. The 50 unsterilized counterparts of these cells had an average capacity of 1.57 $\pm$ 0.10 amp-hr with one cell below 1.00 amp-hr and a range on the remaining cells of 1.40 to 1.75 amp-hr on the formation discharge.

In the first cycle which consisted of a modified c.p. charge and discharge at 0.50 amp, thirteen additional sterilized cells failed to yield 1.00 amp-hr. Each of these had been marginal in the first discharge.

Examination of the records of the components going into each of the sterilized cells, position in the sterilization chamber or during subsequent charging or discharging reveals no pattern which explains the data found on the sterilized cells.

Sufficient separator material was available for sixteen cells which were built and sterilized as before but with the electrolyte level in the cannisters to the tops of the separators. No leakage occurred from the bomb and electrolyte concentration and level was maintained during the sterilization. The average discharge capacity from formation in this group was 2.02 $\pm$ 0.22 amp-hr and the range 1.32 to 2.25 amp-hr. This higher capacity has been maintained through two additional cycles, 1.85 $\pm$ 0.22 amp-hr and 1.81 $\pm$ 0.24 amp-hr respectively. The discharge capacities of both cell groups are given in Table XIII.

TABLE XII
Constructional Details
Three Plate Silver Cadmium Cells

1. Positive Plate

Grid	2/0 Ag
Active Material wt.	6.25 g
Dimensions, inches	1.75 x 1.875 x .028
No. per cell	1
Retainer	1 EM476
Membrane	6 SWRI-GX

2. Negative Plate

Grid	2/0 Ag
Active Material wt.	6.3 g CdO (plus 0.70 g Ni powder)
Dimensions, inches	1.75 x 1.875 x 0.050
No. per cell	2
Absorber	1 EM476

3. Membrane Separator Expansion Factor in 40% KOH
   - Sterilized 1.5 - 1.8
   - Non-sterilized 1.1 - 1.5

   For calculating pack tightness an S.E.F. of 2.5 was used for all cells.

4. Electrolyte - 40% KOH

5. Recombining Electrodes - Silver Electrodes, 1.75 x 1.875 x .010 inches, placed outside of and connected to negative electrodes.

6. Shim Material - Polysulfone sheet .011 and .091 inch

7. Outside Spacer -

Thickness	20 mesh polypropylene screen .046 inch
Placement	Between recombining electrode and shim stock.

TABLE XIII

Discharge Capacities of 3-Plate Silver Cadmium Cells

Cell No.	Non-Sterilized Formation Dischg.[2] Ampere Hours Out[1]	Cycle[3] 1	Cycle[3] 2	Cell No.	Sterilized Formation Dischg.[2] Ampere Hours Out[1]	Cycle[3] 1	Cycle[3] 2
97	1.51	1.63	2.17	42	2.10	1.58	1.58
98	1.51	1.30	1.55	43	1.95	1.84	1.84
99	1.47	1.35	1.60	44	1.33	0.85	0.85
100	1.54	1.33	1.60	45	1.25	1.21	1.39
101	1.48	1.39	1.65	46	1.30	1.30	-
102	1.54	1.65	1.75	47	1.12	0.62	0.80
103	1.43	1.30	1.55	48	1.30	1.49	1.55
104	1.48	1.52	1.60	49	1.19	1.05	1.10
105	1.54	1.38	1.60	50	1.28	0.70	-
106	1.43	1.43	1.43	51	1.15	0.20	-
107	1.47	1.38	1.72	52	0	-	-
108	1.44	1.23	1.52	53	1.00	0	-
109	1.40	1.10	1.23	54	1.33	1.50	1.65
110	1.55	1.40	1.72	55	1.18	1.17	1.25
111	1.45	1.25	1.47	56	1.20	1.02	-
112	1.59	1.58	2.03	57	1.35	1.58	-
113	1.53	1.40	1.79	58	0.63	0.17	0.78
114	1.55	1.44	1.86	59	1.11	1.00	1.00
115	1.40	1.40	1.86	60	1.33	1.52	1.37
116	1.53	1.30	1.71	61	1.10	1.36	1.68
117	1.55	1.30	1.31	62	1.33	1.62	1.70
118	1.50	0.93	1.37	63	1.20	0	-
119	1.59	1.22	1.13	64	1.77	1.87	1.85
120	1.50	1.22	1.08	65	1.53	1.70	1.70
121	1.59	1.17	1.32	66	1.77	1.15	1.50
122	1.53	1.32	1.58	67	1.74	1.91	1.79
123	1.62	1.24	0.53	68	1.74	1.73	1.78
124	1.50	1.26	1.37	69	1.69	1.72	1.73
125	1.55	1.36	1.41	70	1.70	1.91	1.84
126	1.45	0.93	0.70	71	1.60	1.66	1.75
127	1.59	1.20	1.53	72	1.77	1.84	1.79
128	1.73	1.19	1.43	73	0	0.32	0.25
129	1.67	1.33	1.72	74	1.25	1.33	0.84
130	1.68	1.45	1.75	75	1.60	1.65	0.90
131	1.60	1.39	1.50	76	1.45	0.74	1.74
132	0.30	0.15	0.17	77	1.58	0.68	0.85

TABLE XIII

Discharge Capacities of 3-Plate Silver Cadmium Cells

		Ampere Hours Out[1]					
Cell No.	Non-Sterilized Formation Dischg.[2]	Cycle[3] 1	2	Cell No.	Sterilized Formation Dischg.[2]	Cycle[3] 1	2
133	1.60	1.03	1.36	78	1.40	1.60	1.55
134	1.73	1.30	1.50	79	1.60	1.98	1.85
135	1.67	1.45	1.76	80	1.28	1.60	1.93
136	1.68	1.32	1.70	81	1.45	0.87	0.39
137	1.60	1.25	1.20	82	0.38	0.0	-
138	1.65	1.23	1.20	83	1.58	1.80	1.74
139	1.60	1.07	0.75	84	1.23	0.0	-
140	1.68	1.35	1.38	85	1.33	0.25	0.38
141	1.74	1.14	0.82	86	1.55	0.70	0.70
142	1.62	1.20	1.13	87	1.55	0.65	-
143	1.73	1.20	1.38	88	1.23	0.0	-
144	1.70	1.27	1.19	89	0.38	-	-
145	1.68	1.45	1.39	90	2.11	2.13	2.05
146	1.68	1.45	1.39	91	1.32	1.26	1.14
161	1.65	1.59	1.35	92	2.07	2.13	2.03
162	1.80	1.73	1.55	93	2.12	1.90	1.83
163	1.70	1.61	1.43	94	2.08	1.94	1.79
164	1.70	1.76	1.45	95	2.07	1.80	1.89
165	1.85	1.67	1.80	96	2.00	2.14	1.93
166	1.75	1.68	1.50	148	2.04	2.10	2.00
167	1.75	1.56	1.42	149	2.20	1.88	1.85
168	1.78	1.61	1.20	150	2.08	1.99	2.00
169	1.80	1.60	1.20	151	2.18	1.88	1.83
170	1.80	1.59	1.22	152	1.80	1.45	1.65
				153	2.23	1.68	1.65
				154	1.78	1.46	1.45
				155	2.00	1.97	1.95
				156	2.25	1.92	1.91

NOTES (1) Discharge is at 0.50 A to 0.80 volts.

(2) Formation charge at a constant current of .034 amp for 100 hrs.

(3) Recharge each cycle is c.p. at 1.60 v with initial current limited to 0.30 amp.

On the basis of the cycling data obtained on these latter cells, the decision was made to reject the first batch of sterilized cells and re-build a sufficient quantity to fill out the test program and sterilize them immersed in electrolyte to the separator tops. Additional separator material for this purpose has been obtained, plates constructed, and the stacks assembled and sterilized. Resistivity data on the separator material used in constructing the cells is given in Table XIV.

During the next quarter, the electrical characteristics of the cells now being sterilized will be measured, the five cell batteries assembled and sealed, and the test program begun. It is estimated that the cycling, stand, and float experiments will commence at the beginning of August.

TABLE XIV

Electrical Resistivity of Membrane Separators Used in Cell Construction

Lot No.	Sample	Wet Thickness cm. x 10^3	Resistance in 40% KOH Ω-cm^2	avg.
SWRI GX28	1	5.4	0.13	
	2	5.4	0.15	
	3	5.4	0.31	0.24
	4	4.1	0.30	
	5	4.1	0.31	
SWRI GX31	1	3.9	0.42	
	2	3.7	0.48	
	3	3.3	0.28	0.28
	4	3.5	0.12	
	5	4.1	0.10	
SWRI GX34	1	4.1	0.12	
	2	4.1	0.11	
	3	4.1	0.16	0.13
	4	4.1	0.11	
	5	3.9	0.11	
SWRI GX21 (sterilized)	1	5.6	0.09	
	2	5.9	0.09	
	3	6.8	0.14	0.14
	4	6.5	0.24	
	5	6.8	0.15	
SWRI GX21	1	4.3	0.22	
	2	3.8	0.15	
	3	4.1	0.13	0.15
	4	4.9	0.14	
	5	4.6	0.10	

Experimental studies during the second quarter of 1967 were directed to examination of a new polyphenylene oxide modification, evaluation of a new terminal design, demonstration of epoxy seals on cell cases, and attempts to effect hot gas welds on PPO 541-801.

I. GLASS FILLED POLYPHENYLENE OXIDE

The comparative physical properties of the glass filled (30 wt. %) GFP3-111 relative to PPO 531-801 and PPO 541-801 are given in Table XV. The significant properties of GFP3-111 are: the specific gravity (1.27), tensile strength at 257°F (15,500 psi), and coefficient of linear expansion (1.5×10^{-5} in./in./°F). These properties are advantageous with respect to PPO 531-801 or PPO 541-801 and justify preliminary screening evaluations on the glass filled material.

ASTM tensile bars were used in preliminary evaluations of GFP3-111 shown in Table XVI. The initial tensile strength was 16,500 psi. After 35 hr. in saturated steam at 135°C, the tensile strength of bent tensile bars decreased to 10,200 psi although there were no craze cracks visible at magnifications of 14 to 60. The specimens had an average weight increase of 0.32%, and no significant dimensional or hardness change in this steam crazing test.

For the sterilization test, the tensile bars were partially immersed in 40 wt. % KOH and held at 135°C for 35 hr. The specimens had an average weight loss of 0.38 wt. % and a white matte surface of raised glass fibers. The tensile strength decreased to 12,400 psi.

A butt weld was prepared in a 4 in. disk; the disk was cut into 1/2 in. strips for tensile tests. The average tensile strength was 3,000 psi which is not encouraging. Further studies on this material can not be justified in the time available.

II. MOLDED CASE ASSEMBLIES - NEW TERMINAL DESIGN

Fine silver terminals, Drawing No. 1-096 (Fig. 3 in Report for First Quarter 1967), were turned by Artcraft Machine Company, Langhorne, Pa. One hundred terminals were checked with gauges for length and outside diameter. The inside diameter ($0.125 \begin{smallmatrix}+0.003\\-0.000\end{smallmatrix}$) on all terminals was checked with a plug gauge since this dimension is critical for fitting into the cover mold. A few of the terminals were reamed with 0.125 in. reamer to remove a burr. All dimensions on the terminals were within specified tolerances.

TABLE XV
Properties of Polyphenylene Oxides

	ASTM Method	PPO531-801	PPO541-801	GFP3-111
Specific Gravity	D 792	1.06	1.43	1.27
Water Absorption Equilibrium				
73°F (wt. %)		0.10	0.12	0.10
212°F (")		-	0.31	-
Tensile Strength Yield	D 638			
73°F (psi)		10,000-11,000	12,500	18,000
257°F (")		5,500- 6,500	-	15,500
Tensile Modulus	D 638			
73°F (psi)		3.7×10^5	7.4×10^5	1.24×10^6
257°F (")		3.3×10^5	-	1.20×10^6
Heat Deflection at 264 psi (°F)	D 648	375-380	337	360
Coefficient of Linear Expansion (in./in./°F)	D 696	2.9×10^{-5}	-	1.5×10^{-5}
Mold Shrinkage in./in.	D 955	0.007	0.003	0.001

Data from General Electric Company literature.

TABLE XVI
Properties of GFP3-111 (Glass filled PPO)

As Molded	Maximum psi	Minimum psi	Average psi
Tensile strength	16,600	16,400	16,500
After Steam Crazing Test (35 hr. 135°C saturated steam, 1/4 in. deflection between 5 3/4 in. centers)			
Tensile strength	10,800	9,700	10,200
After Sterilization (35 hr. 135°C 50% immersion in 40 wt. % KOH)			
Tensile strength	13,700	11,000	12,400
Appearance	White matte surface; fibers lifted from surface.		
Welded Butt Joint			
Tensile Strength	3,600	2,400	3,000

JPL case assemblies with the terminal Drawing No. 1-096 were molded in PPO 531-801 and PPO 541-801 with JPL source inspection. The molding conditions are given in Table XVII. The case molding required 25-30°F higher molding machine temperatures than the previous lot of PPO 531-801; the cover molding required 10-15°F higher temperatures. The surface finish on the present molding was equal to or better than the previous lot. The molding machine temperatures for cases in PPO 541-801 were also higher on the present lot of moldings. These differences may arise from running on a different molding machine or from errors on temperature indicators.

The variations in molded cases in PPO 531-801 and PPO 541-801 are given in Table XVIII. The dimensional variations for both materials represented the normal spread expected. The weights of the cases showed less than 0.5% difference between the average and either the maximum or the minimum. The variations on the covers are given in Table XIX. The spread in weights of the covers represented good packing in the molding operation. The dimensional variations were characteristic of a good molding operation. The covers were examined under 14 power microscope at the terminal-resin junction for stress cracks.

Twenty covers in 531-801 had very small stress cracks only at 3 positive terminals. Re-examination of these covers 2 weeks after the molding revealed 4 additional terminals with very small stress cracks. These results represented an improvement over the previous terminal. Six weeks later, however, all 20 covers showed some stress cracks usually 1-2 mils long. There was no significant crack propagation, however, in this time. The new terminal design has reduced crack propagation but has not eliminated the problem. A coating of the resin-terminal junction on top and bottom of the cover with an epoxy resin appears to be required for the molded terminal insert.

The covers molded in PPO 541-801 had no stress cracks at the terminal at the outset and developed none in six weeks, presumably due to the lower coefficient of thermal expansion of this filled resin.

III. EPOXY SEALS

A. PPO 531-801 Cases and Covers

A lot of 8 JPL cases (Figure 19, 24, 25 with silver terminal 1-096) in PPO 531-801 were prepared to show reproducibility of the epoxy seal through pressure tests, sterilization procedure and final helium leak rate. The total results are given in Table XX which show 7 out of 8 cases passing required tests. The pressure loss during sterilization was 9 to 11 psig; the weight loss was 2.5 to 3.4 g. The final helium leak rate was 7.5×10^{-9} to 1.2×10^{-6} ml/sec with one case at 8.7×10^{-6} ml/sec. These results

TABLE XVII

Molding Conditions for JPL Cell Case Assemblies with Silver Terminals (Drawing No. 1-096)

Molder	S & T Tool & Die Co., 125 E. Railroad Ave., Monrovia, California			
Machine	Reed Prentice, Model 200 TD, 9 oz, 200 ton clamp			
Material	PPO 531-801 Lot 0093		PPO 541-801 Blend 007	
	Case	Cover	Case	Cover
Cylinder Temperatures (°F)				
Feed	560	540	568	540
Center	580	560	605	580
Nozzle	605	595	600	590
Melt	625	610	610	610
Front	580	575	615	590
Mold Temperatures (°F)				
Core	290-330	-	310-340	-
Mold	280	270	285	270
Pressure (psig)				
Inject	1800	1600	1300	2000
Hold	1200	1200	1200	1900
Cycle (sec)				
Fill	4-5	1	3	2
Inject	30	30	30	30
Cool	50	15	40	15
Total (approximate)	90	58	95	58
Molding Date	5/17/67	5/17/67	5/17/67	5/17/67

JPL Source Inspection

TABLE XVIII
Variations in Molded JPL Cell Cases

Material	PPO 531-801	PPO 541-801
Specimens	20	20
Hardness Average (Shore D)	85	90
Weight (g)		
Maximum	112.12	150.00
Minimum	111.50	149.38
Average	111.90	149.70
Outside Dimensions (in.)		
Length (at top)		
Maximum	3.180	3.193
Minimum	3.175	3.187
Average	3.178	3.190
Width (at top)		
Maximum	1.004	1.009
Minimum	1.000	1.003
Average	1.002	1.007
Height		
Maximum	6.068	6.086
Minimum	6.062	6.080
Average	6.067	6.082
Inside Dimensions at top (in.)		
Length		
Maximum	2.930	2.933
Minimum	2.927	2.930
Average	2.928	2.931
Width		
Maximum	0.760	0.762
Minimum	.750	.758
Average	.756	.760

Weights $\pm$ 0.02 g

Dimensions $\pm$ 0.001 in.

TABLE XIX
Variations in Molded JPL Cell Covers wtih
Terminal Drawing No. 1-096

Material	PPO 531-801	PPO 541-801
Specimens	20	20
Hardness Average (Shore D)	85	90
Weight (g)		
Maximum	21.68	26.13
Minimum	21.53	26.10
Average	21.62	26.07
Dimensions (in.)		
Top length		
Maximum	2.941	2.950
Minimum	2.938	2.947
Average	2.940	2.949
Top width		
Maximum	0.775	0.779
Minimum	.774	.774
Average	.775	.778
Bottom length		
Maximum	2.935	2.947
Minimum	2.933	2.943
Average	2.934	2.944
Bottom width		
Maximum	0.767	0.771
Minimum	.766	.769
Average	.767	.769
Stress Cracks	Very small, at 3 positive terminals.	None

Weights $\pm$ 0.02 g

Dimensions $\pm$ 0.001 in.

X Microscope 14X

TABLE XX

Adhesive Seals on JPL Cases in PPO 531-801

Cases	8
Seal Design	Top and bottom edges of cover 1/32 in. chamfer, inside top edge of case 1/32 in. chamfer. All mating surfaces sanded 240 emery cloth. Mating surfaces and top and bottom junctions terminal with resin painted with resin.
Resin	Dow DEN 438-EK85 10.0 g Rohm & Haas DMP 30 0.42
Cure	64 hr. at 25 ± 2°C 4 " " 50 " 4 " " 75 "
Pressure Test (90 psig 25°C)	8 Pass

Sterilization

(65 psig 120 hr. 135°C)

Case No.	1	2	3	4	5	6	7	8
Total pressure loss (psig)	10	11	10	10	9	9	10	11
Weight loss (g)	3.4	3.3	3.2	3.0	2.5	2.5	2.6	2.6
Helium leak rate (ml/sec)*	1.2×10^{-6}	1.7×10^{-8}	3.4×10^{-8}	1.0×10^{-7}	8.7×10^{-6}	$< 7.5\times10^{-9}$	7.5×10^{-7}	3.0×10^{-8}

Electrolyte Slurry

Each case

KOH 40 wt. %	50 ml
ZnO	5.6 g
Ag powder	0.1

* ESB-EMED Inspection Specification 2.3.3.3 Fig. 2.10

demonstrate feasibility of the epoxy seal on JPL cases, although it was necessary to paint the top and bottom of the covers around the terminal inserts with epoxy to insure against possible bad effects due to the stress cracks in the covers.

B. PPO 531-801 Cases and PPO 541-801 Covers

PPO 541-801 covers have never shown any stress cracks at the molded insert terminal which is an advantage over PPO 531-801. An attempt was made to seal a cover of PPO 541-801 into a case of PPO 531-801 to retain the advantages of a molded terminal insert. The results on 12 cases for this evaluation are given in Table XXI. All 12 cases sealed with the epoxy resin (Dow DEN438-EK85) passed the 90 psig test at room temperature, but only 2 out of 7 cases sustained 65 psig at 135°C. The differential thermal expansion along the longer dimension of the top amounted to 0.007 in. This added stress was probably responsible for the seal failures. Based on these results the combination of a cover in PPO 541-801 sealed into a PPO 531-801 case by an epoxy resin does not appear feasible. The thermal effect alone was evaluated by heating the remaining 5 cases to 135°C for 16 hr; after this thermal cycle all 5 cases sustained 90 psig at room temperature.

C. Sterilization of Adhesive Epocast 221 on PPO 531-801

Joints were prepared on PPO 531-801 ASTM tensile bars to evaluate the stability of this adhesive under sterilization conditions. The tensile bars were cut in the center of the neck area, sanded and painted with the adhesive. The cure cycle and bond strengths are given in Table XXII, which shows a decrease in bond strength from 1050 psi to 850 psi after sterilization. All failures were of an adhesive type - the epoxy separated cleanly from the surface of PPO 531-801. Thus, this resin is not considered useful as an adhesive to PPO 531-801.

IV. WELDED JPL CASES IN PPO 541-801

Difficulties have been encountered in hot gas welding JPL cases in PPO 541-801; usually there were leaks in the welds which were found in the pressure tests. The PPO 681-111 has been found to weld easily; hence this material was used as a welding rod. A butt weld was prepared in a 4 in. disk; the disk was cut into strips 1/2 in. wide which were pulled. The tensile strengths were: maximum 3,800 psi, minimum 2,300 psi and average 3,200 psi. The results on 5 cases in PPO 541-801 welded with rod of PPO 681-111 are given in Table XXIII. Four of 5 cases passed the 90 psig test but failed at 135°C within a few hours at 65 psig. These results indicate that a combination of resins in the weld holds no promise of a satisfactory seal.

TABLE XXI
Adhesive Seals on JPL Cases in PPO 531-801
with Covers in PPO 541-801

Cases	12
Seal Design	Covers individually fit to cases, inside top edge of case and all top and bottom edges of cover chamfered 1/32 in. All mating surfaces sanded 240 emery cloth and painted with resin.
Resin	DEN438-EK85 10.0 g DMP30 0.42
Cure	70 hr. at 25 ± 2°C 4 " " 50 " 4 " " 75 "
Pressure Test (90 psig 25°C)	12 Pass
Sterilization Pressure (65 psig 120 hr. 135°C)	2 Pass 5 Fail Failures on short side of cases

TABLE XXII

Sterilization of Adhesive Joints (Epocast 221 + 8 wt. % Hardener 927) on PPO 531-801

Specimens	ASTM tensile bars cut in center
Formulation	Epocast 221 5.0 g (Furane Plastics) Hardener 927 0.40
Joint	1/2 in. x 1/4 in. sanded with 240 emery cloth painted with adhesive.
Cure	16 hr. at 25°C under 4 psi 2 " " 75°C no pressure 3 " " 145°C " "

Bond Strength

	Control	After Sterilization (1)
Samples	3	4
Maximum	1100 psi	1100
Minimum	1000 "	750
Average	1050 "	850
Failures	Adhesive	Adhesive

(1) Sterilization: 113 hr. at 135°C
Joints immersed in electrolyte slurry

KOH (87 wt. %)	530 g
Hn	690
ZnO	80
Ag powder	0.5

Parr Autoclave 2000 ml 316 SS

Temperatures ± 2°C
Weights ± 0.02 g
Tensile ± 100 psig

TABLE XXIII

Welded Seals on JPL Cases in PPO 541-801 Using PPO 681-111 Welding Rod

Cases	5 Top edges of case and cover chamfered to form 60° "V" 1/8 in. deep.
Drying	Cases and rod dried 3 hr. at 110° ± 2°C.
Welding	Cover held in case by rectangular camp 1/2 in. from top of case.

	N_2 psig	Voltage v	Amp
Preheating gun	6	112	-
Welding gun	6	115	7.4

Annealing	2 min with guns
Pressure Test (90 psig 25°C)	4 Pass 1 leak in weld
Sterilization (65 psig 120 hr. 135°C)	4 Fail

Welded JPL cases in PPO 541-801 with the silver terminal (Drawing No. 1-096) were prepared with 1/8 in. round welding rod obtained by cutting sections from a molded case of the same material. The welding conditions and results are given in Table XXIV. The preheating was continued until the bottom of the V's began to fuse then welding rod was applied. Only 2 welds out of 10 passed the 90 psig test. Leaks in welds were repaired by milling a slot and welding again. This operation was a failure since 8 out of 8 welds still leaked. These results indicate that a feasible welding operation has not been demonstrated, and a welded seal on PPO 541-801 cases can not be recommended.

TABLE XXIV
Welded Seals in JPL Cases in PPO 541-801

Cases	10 Top edges of cases and covers chamfered to form 60° "V" 1/8 in. deep.
Drying	2 hr. at 120 ± 2°C
Welding	Cover held in case by rectangular clamp 1/2 in. from top of case.

	N_2 psig	Voltage v	Amp.
Preheating gun	6	114	-
Welding gun	6	120	7.6

Pressure Test (90 psig, 25°C)	2 Pass 8 weld leaks
	After repairing 8 weld leaks, all 8 cases still failed 90 psig test due weld leaks.

FABRICATION AND TESTING OF CELLS

1. HIGH IMPACT TESTS ON MODEL 334 HEAT STERILIZABLE CELLS

Design objectives for the Model 334 sealed ZnO/KOH/Ag cell were:

- Capability of heat sterilization for 120 hours at 135°C before formation charge.
- Charge, float for 8 months, then survive a planet landing shock estimated to be 10,000 g peak for 0.1 msec. followed by 5,000 g for 3 msec.
- Discharge at mission rates delivering 50 AH above 1.30 volts per cell.

Cell design features were described in detail in Reference (1). The Model 334 cell featured all plastic plate supports to yield maximum energy per unit weight. Severe constraints were imposed by limited choice of materials (polyphenylene oxide and polysulfone), inability to mold plate support struts in thin plate cross-sections, required use of existing jar molds, and the discovery late in cell development that the plate support plastic (PPO 681-111) exhibited only 70% of its virgin tensile strength after being heat pressed onto plate grid structures.

Thirteen cells were manufactured. Six were submitted to a heat sterilization test of 120 hours at 135°C, then cycled through two 100% depth discharge cycles and shocked. Three cells were shocked with no prior heat sterilization, and three cells were cycled with no heat sterilization or shock. One control cell was dissected after shorting on the first charge because of a manufacturing defect.

Table XXV summarizes cell weights, impact velocities, and stopping distances observed during impact testing at the Jet Propulsion Laboratory, Pasadena, California. Peak "g" levels were measured with accelerometers mounted on the cell shock fixture. The calculated "g" levels for cells S/N 5, 10, and 12 all of which were heat sterilized 120 hours were 4,020 to 4,180 "g". Cells S/N 2, 3, and 4 also heat sterilized were shocked at higher velocities and reached calculated "g" levels from 7,420 to 7,500 "g". Cells S/N 6, 7, and 8, the controls with no heat sterilization, were given the maximum shock reaching 7,900 to 8,200 mean "g" for 0.4 msec. and 10,000 peak "g".

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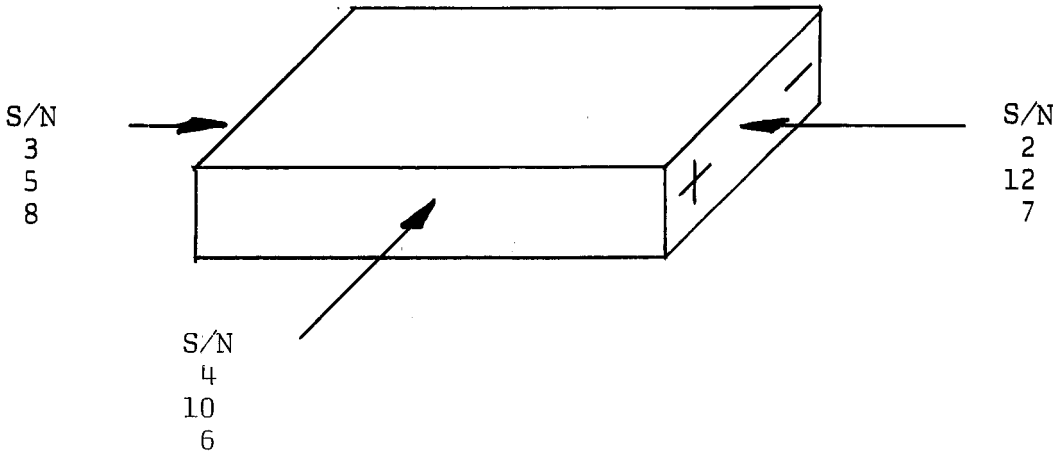
(1) First Quarterly Report, Heat Sterilizable, Impact Resistant Cell Development, May 9, 1967, p. 40-49.

TABLE XXV

SHOCK DATA FOR MODEL 334 CELLS

S/N	Weight (gms)	Impact Velocity (Ft/Sec)	Tool Diameter (in.)	Stopping Distance (in.)	Pulse Duration (Msec.)	Calculated Avg. "G"	Measured Peak "G"	HS * hrs
5	788	109	3/4	0.530	0.8	4020	6000	120
10	775	110	3/4	0.543	--	4140	----	120
12	788	110	3/4	0.538	0.8	4180	5000	100
2	784	173	1	0.751	0.5	7420	9000	36
3	795	172	1	0.737	0.5	7500	9500	120
4	820	173	1	0.751	0.5	7420	9000	120
6	805	150	1-1/8	0.515	0.4	8150	10000	0
7	790	150	1-1/8	0.532	0.4	7900	10000	0
8	800	150	1-1/8	0.511	0.4	8200	10000	0

VELOCITY VECTORS AT SHOCK



*Heat sterilization hours at 135°C before formation charge and high impact.

Table XXVI summarizes loaded voltage tests performed at discharge rates of 2, 10, 20, 30, and 40 amps before and after high impact with the calculated voltage decreases. Figure 1 shows that the decrease in loaded voltage was greater at increasing "g" values of shock and increasing currents of discharge.

Cells 5, 10, and 12 shocked at 4,100 "g" after 120 hours heat sterilization maintained good loaded voltage and only one of three - S/N 12 - suffered a seal failure. These three cells were left at JPL to undergo post-shock cycling.

All six cells shocked at 7,420 - 8,200 "g" had cell jar failures ranging from corner hairline cracks in one cell to complete wall fracture in the worst case. Failure of jar walls in two cells - S/N 3 and 4 - during terminals forward shocks left the subcovers inadequately supported which also failed. Flexing of jar walls in four cells damaged the epoxy seal which failed in peel.

Plate strut damage was severe. One shocked cell - S/N 7, an unsterilized cell - showed 60% undamaged plates. In the other five cells which were dissected all struts were broken or damaged. Terminal forward shock damaged plates not adequately supported in buckling despite the use of epoxy shims designed to perform this function. The tensile modulus of PPO 681-111 after heat pressing operations is too low to prevent buckling when case walls bulge outward during impact.

Extensive wrinkling and breakage of charged positive active material (AgO) indicated that greater support from grid structures in the plate are needed to survive the maximum shock loading.

As in the Model 325 cell tests plastic strut projections at each bottom corner of the positive plates failed in shear during terminals-forward shocks. Buckling of the struts at the top of each plate shifted too large a shear force to the bottom supports.

Negative plate "M" frames suffered damage in the upper struts associated with delamination of the heat bonded sandwich of two outer PPO 681-111 "M" frames and the inner 35 x 40 mesh Ag screen. PPO 681-111 is the only grade PPO which can be heat pressed and even this grade gives inadequate heat bond strength. The bond between zinc active material and Ag screens in the negatives generally was not broken by the shock.

Polypropylene plate absorbers were not damaged structurally by heat sterilization or shock.

RAI-116 semipermeable membranes were damaged by heat sterilization and by shock. Large splits in single layers were found in sterilized cells - some splits extending from the top of the separator to the bottom of the "U" fold in

TABLE XXVI

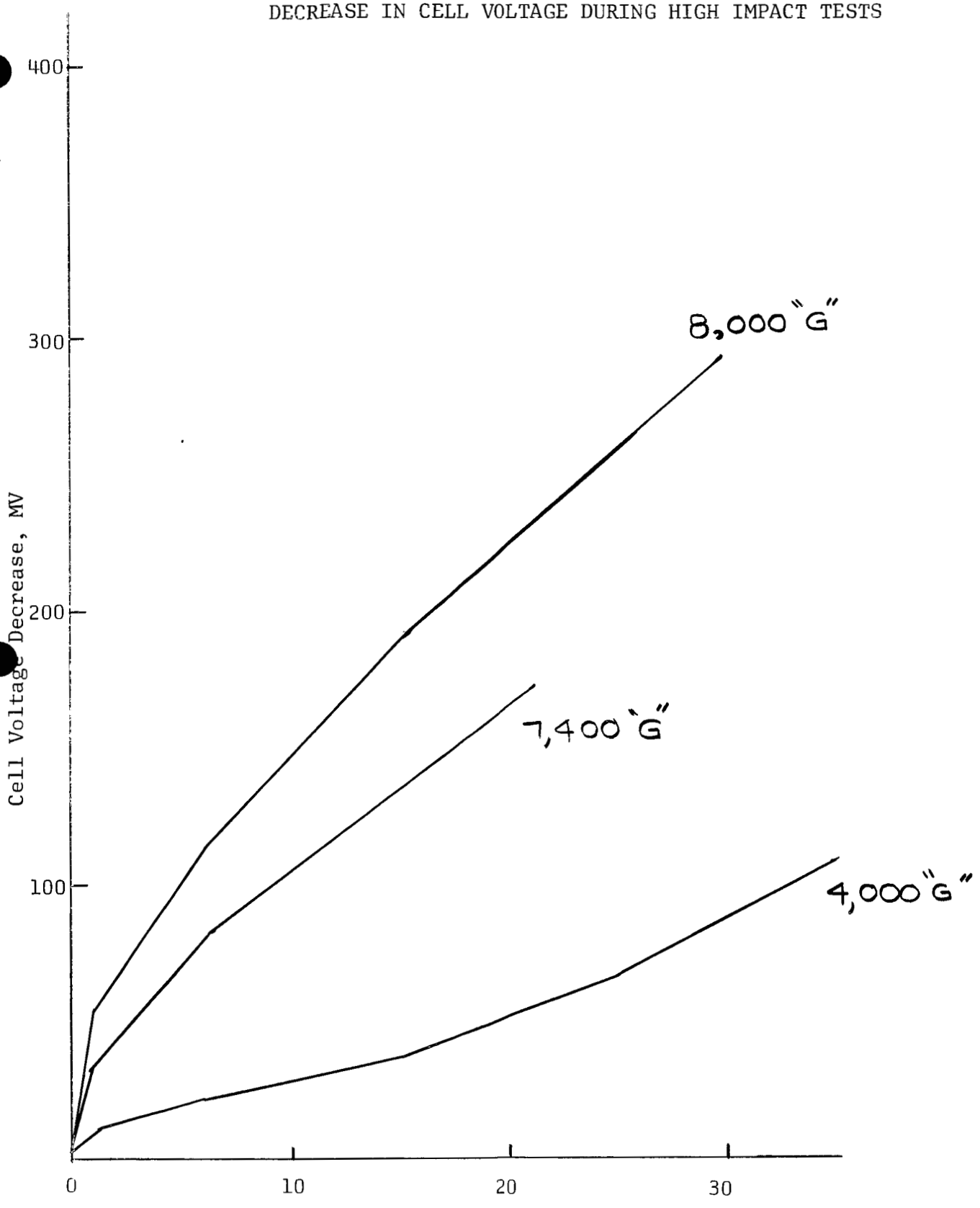
DECREASE IN CELL VOLTAGES FROM HIGH IMPACT

Discharge		4,000 "g"			7,400 "g"			8,000 "g"		
Rate, Amps	Time	5*	10	12	2	3	4	6	7	8
0	Before	1.859	1.859	1.863	1.857	1.860	1.858	1.572	1.860	1.603
	After	1.857	1.855	1.860	1.853	1.855	1.854	1.569	1.859	1.592
	Decrease, MV	2	4	3	4	5	4	3	1	11
2	Before	1.823	1.652	1.831	1.737	1.827	1.825	1.505	1.744	1.482
	After	1.820	1.618	1.820	1.575	1.826	1.821	1.472	1.695	1.288
	Decrease, MV	3	34	11	162	1	4	33	49	194
10	Before	1.738	1.401	1.747	1.574	1.750	1.751	1.280	1.530	1.145
	After	1.719	1.380	1.701	1.273	1.749	1.740	-	1.379	1.028
	Decrease MV	19	21	46	301	1	9	-	151	117
20	Before	1.639	1.200	1.652	1.464	1.667	1.671	.820	1.347	1.028
	After	1.618	1.210	1.585	.998	1.662	1.649	-	1.120	.770
	Decrease, MV	21	-10	67	466	5	22	-	227	258
30	Before	1.478	.998	1.490	1.248	1.480	1.503	-	1.149	-
	After	1.396	.960	1.344	-	1.433	1.438	-	.870	-
	Decrease, MV	82	38	146	-	47	65	-	279	-
40	Before	1.356	-	1.382	1.150	1.397	1.394	-	1.003	-
	After	1.306	-	1.187	-	1.320	1.309	-	-	-
	Decrease, MV	50	-	195	-	77	85	-	-	-

(*) Serial number of cell tested.

FIGURE 1

DECREASE IN CELL VOLTAGE DURING HIGH IMPACT TESTS



the center of some plates. In the Model 334 cell design, inadequate allowance was made for membrane shrinkage. Shrinkage of 0.075 x 2 for plate width of 2.88 will be increased in future cells to 8.7% of plate width. During terminals-aft shocks the rounded bottom edges of positive plates resting on the "U" fold cut into the membrane at points of greatest impact.

Electrochemical performance of the Model 334 cell was below expectations in voltage and capacity and very variable from cell to cell. A prime reason was accumulation of plate thickness tolerances which gave a tight cell pack. Performance is summarized in Table XXVII for control cells S/N 9 and 13 having neither shock nor heat sterilization and in Table XXVIII for the seven heat sterilized cells before shock.

Relative performance on the two rate discharge during two cycles may be summarized as follows:

Test Parameter		Unit	Non-Sterilized Cells n = 2	Wet Heat Sterilized Cells n = 7
Formation	Charge Input	AH	49	54
	Discharge Output	AH	38	40
Cycle 1	Input	AH	44	53
1	Output	AH	42	41
1	Energy	WH	59	58
1	Energy	WH	34	33
	Density	lb.		

The differences in the above means are well within the variability of the individual cells in either group. A. C. impedances measured between individual cell terminals with a Keithley Milliohmmeter at 90 Hz revealed one cause of variability:

Time of Measurement	A. C. Impedance-mohms			
	X	n	Min.	Max.
At seal	59	5	40	130
After heat sterilization	23	7	6	44
After formation discharge	12	7	1	22
After Cycle 1 discharge	17	7	12	24
After Cycle 2 charge	31	7	5	90

TABLF XXVII

MODEL 334 CONTROL CELL CYCLING TESTS

Test Parameter	Cycling Characteristics S/N 13 1	S/N 13 2	S/N 13 3	S/N 13 4	S/N 9 1	S/N 9 2	S/N 9 3	S/N 9 4	S/N 9 5	S/N 9 6	S/N 9 7	Mean All
1.0 Charge Profile	(1)	(2)	(6)	(6)	(2)	(2)	(2)	(2)	(2)	(2)	(6)	
1.1 Acceptance (AH)	47.9	40.0	50.0	30.6	51.7	48.6	44.9	38.1	41.6	38.3	31.3	
1.2 Efficiency (%)	(4)55	122.	137.	111	(4)59	110	93	101	101	88	89	
1.3 AC Impedance (mohm)												
Before Charge	4.4	44	26	30	150	8	0.2	1.8	2.6	13	--	
After Charge	8.2	43	40	NR	18	2	3	1.7	6.3	32	49	
1.4 Cell Pressure (psig)												
Start Charge	-5.5	0	7	6	-12.5	1.5	-2.5	-3	-2	-3.2	-2.5	
End Charge	30	7	15.5	8	7.5	3	5.5	2.5	5	2.5	3.0	
2.0 Days Charged Stand (7)	2	11	25	26	1	1	5	2	8	8	33	
2.1 Discharge Temp. °C Profile (3)	25	25	25	25	25	25	0	25	65	25	25	
2.2 Capacity (AH)							(5)					
@10 amp to 1.30V	10.7	17.5	8.2	13.2	35.0	35.0	28.3	32.7	39.3	28.3	27.5	28.3
@ 2 amp to 1.30V	22.1	18.9	19.2	8.8	9.3	13.2	9.3	8.6	4.1	6.6	9.0	11.2
Total	32.8	36.4	27.4	22.0	44.3	48.2	37.6	41.3	43.4	34.9	36.5	39.5
2.3 Efficiency (% Previous Charge)	73	91	54	72	85	99	84	107	104	91	116	94
2.4 Voltage (Volts)												
10 amp plateau	1.30	1.32	1.32	1.305	1.39	1.405	1.275	1.38	1.46	1.42	1.40	1.37
average	1.358	1.397	1.356	1.335	1.413	1.425	1.275	1.399	1.462	1.42	1.38	1.40
2 amp peak	1.46	1.45	1.44	1.47	1.48	1.48	1.43	1.50	1.44	1.45	1.44	1.46
average	1.413	1.414	1.414	1.419	1.429	1.413	1.309	1.418	1.402	1.420	1.403	1.40
2.5 Energy (WH)	45.7	50.3	38.2	30.1	62.7	68.5	48.2	58.0	63.2	49.6	50.6	55.8
2.6 Energy Density (WH/lb.)	26.3	28.9	22.0	17.3	36.5	39.8	28.0	33.7	36.8	28.8	29.4	32.4
2.7 AC Impedance (mohm)												
Before Discharge	8.2	--	90	190	18	3	--	5	1.9	27	210	
After Discharge	22	--	40	NR	8.4	1	--	2.5	5.8	15	180	
2.8 Cell Pressure (psig)												
Start Discharge	2	10	5.5	NR	1.5	0	--	-1.2	7	-2	-1.7	
Peak Discharge	21	7.5	6.5	NR	3	0	--	-2	10	-2	0	
3.0 Net Input (AH)	--	55.1	68.7		--	56.0	52.0	53.2	53.5	48.4	44.8	54.0

(1) Charge Profile (cc) I = 0.5A to lower plateau V; I = 1A to 1.97; I = 0.5A to 1.97V.
(2) Charge Profile (cc) I = 1A to 1.97 V; I = 0.5A to 1.97 V at 25°C.
(3) Discharge Profile: I = 10A to 1.30V; I = 2A to 1.30 V (5 sec. pulses at 2, 10, 20, 30 & 40 amp at start & 90 minutes discharge time).
(4) Formation Charge Efficiency = % of theoretical Ag capacity.
(5) Capacity to 1.20 V.
(6) CP Charge 1.95 ± .01 V; I = 1.5A maximum; T = 72 hrs. maximum.
(7) Charged wet stand at 25°C.

TABLE XXVIII

ELECTRICAL TESTS ON MODEL 334 CELLS BEFORE AND AFTER HEAT STERILIZATION

Test Parameter	Unit	S/N 2	3	4	5	10	11	12	Mean	Notes
1. A.C. Impedance at Seal	mohm	--	40	--	40	47	130	40	59	
2. Heat Sterilization Time	hrs	34L	120	120	120	120	16L	100L		(5)
3. A.C. Impedance After HS	mohm	8.8	16.5	5.9	16.5	25	40	44	23	
4. Formation Charge Input	AH	61.3	51.5	51.5	57.7	52.2	51.8	52.1	54.0	(1), (2), (3)
5. Formation Discharge Output	AH	49.2	42.7	36.5	39.7	35.8	34.4	42.1	40.1	(4), at R.T.
6. A.C. Impedance Before Charge	mohm	22	16	13	2	2	21	1	12	
7. Cycle 1 Input 1.95V CP	AH	55.7	--	47.4	48.7	54.4	55.8	54.9	52.8	(3)
8. Cycle 1 Output, Total	AH	33.0	40.7	42.5	39.9	40.5	39.7	47.5	40.5	(4) at R.T
At 10A to 1.30V/C		30.5	34.2	37.8	36.9	13.5	30.2	40.8	32.0	
At 2A to 1.30V/C		2.5	6.5	4.7	3.0	27.0	9.5	6.7	8.6	
9. Cycle 1 Plateau Voltage	volts									
At 10A		1.42	1.43	1.42	1.42	1.37	1.39	1.43	1.41	
At 2A		1.51	1.52	1.56	1.42	1.45	1.47	1.46	1.48	
10. Cycle 1 Engergy Output	WHr	46.3	59.9	--	58.3	56.6	55.8	68.7	57.6	
11. Cycle 1 Energy Density	WH/lb	26.8	33.8	--	33.1	32.3	30.7	39.0	32.6	
12. A.C. Impedance After Cycle 1	mohm	23	13	18	16	24	12	13	17	
13. Cycle 2 Input 1.95V CP	AH	76.2	39.6	40.8	43.7	40.8	41.1	47.0	47.0	
14. A.C. Impedance After Change	mohm	38	24	15	5	90	17	25	31	
15. Net Input	AH	111.0	--	60.7	70.5	71.1	74.6	64.4	75.3	(6)

Notes: (1) Constant current: 0.5A to lower plateau; 1.0A to 1.97; 0.5A to 1.97A at 25°C.
(2) Constant current: 1.0A to 1.97V; 0.5A to 1.97V at 25°C.
(3) CP (modified) : initial current limited to 1.5A; voltage at 1.95±.01V.
(4) Constant current: 10A to 1.30V; 2A to 1.30V.
(5) L=KOH leaks at seal
(6) Cell S/N 2 went into full float on CP charge.

CONCLUSIONS

- High impact tests at peak values of 10,000 "g" severely damaged Model 334 plates reinforced with PPO 681-111 plastic frames and struts.

- Cell cases were fractured by bulging cell packs moving under impact stresses.

- Extensive wrinkling and breakage of AgO active material in positive plates demands that future plate structures be reinforced with metals to achieve adequate design safety factors.

- RAI-116 membranes in the separator system were split in single layers by the combination of shrinkage during heat sterilization and high impact.

II. DEVELOPMENT OF "E" DESIGN MODEL 343 CELLS

Thirteen Model 343 cells, the successor to the Model 334, are in final stages of production in the Engineering Pilot Plant. Design objectives were:

- Cell jar walls and cover of increased thickness and strength to eliminate jar failure during shock.

- Plate structures reinforced with non-magnetic metal struts rather than plastic to prevent fracture of plates during shock.

- Use of a new membrane to decrease performance variability.

- Nominal capacity of 25 AH and performance per JPL Specification GMP-50437-DSN-C.

Additional design constraints which compromised engineering decisions were the required use of JPL jar (Reference 2) with cell height as the sole variable and uncertain allowances for separator wet thickness and capacity losses during heat sterilization and shock.

Table XXIX summarizes the materials, weights, and major dimensions for the cell components. For an output of 25 AH at the 2 hour rate at an operating voltage of 1.39 volts per cell and a cell weight of 1.26 pounds the design energy density is 27 WH/lb. and 2.1 WH/in^3. Figure 2 is an assembly sketch of the Model 343 cell.

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(2) Annual Report, Heat Sterilizable, Impact Resistant Cell Development, April 21, 1967, p. 67.

TABLE XXIX

MODEL 343 25 AH
HEAT STERILIZABLE HIGH IMPACT CELL DESIGN

CELL COMPONENT	NO IN CELL	MATERIAL	DIMENSIONS, INCHES				WT. PER CELL gms
			L	W	H	T	
Cell Case	1	PPO-531-801	1.00	3.15	4.52	.125	83.3
Subcover	1	PPO-531-801	2.88	.74	.50	.125	13.6
Top Cover	1	PPO-531-801	3.15	1.00	.50	.125	11.8
Side Shims	2	PPO-531-801	4.03	.72		.100	12.6
Sealing Tape	1	Epoxy-glass		.50		.00	10.
Cement	2	Epoxy					15.
	bulk						
Sealant	2	Epoxy					20.
	bulk						
CellPack							
Positive Blanks	4	Porous Ag	3.40	2.45		.055	89.2
Positive Grids	4	Titanium	3.40	2.45		.008	28.4
Positive Absorber	4	EM-476 (IL)	8.06	4.96		.003	3.6
Positive Membrane	4	SWRI-GX (5L)	28.0	8.00		.003	20.5
Negative Retainer	5	EM-476 (IL)				.003	3.0
Center Negative Active Material	3	ZnO	3.42	2.55		.079	59.8
Center Grids	3	Ag	3.40	2.53		.008	41.4
End Negative Active Material	2	ZnO	3.42	2.55		.053	19.8
End Negative Grids	2	Ag	3.40	2.53		.008	27.6
Electrolyte, 68 cc	bulk	40% KOH					95.
Hardware (Terminals, hex nuts, and washers)							16.
Cell Dimensions							
Overall			1.14	3.29	5.69	-	
Case Only			1.00	3.15	5.18	.125	
Cell Weight, Wet, Sealed							570. (1.26 lbs)
Cell Volume, in^3 (16.3)							

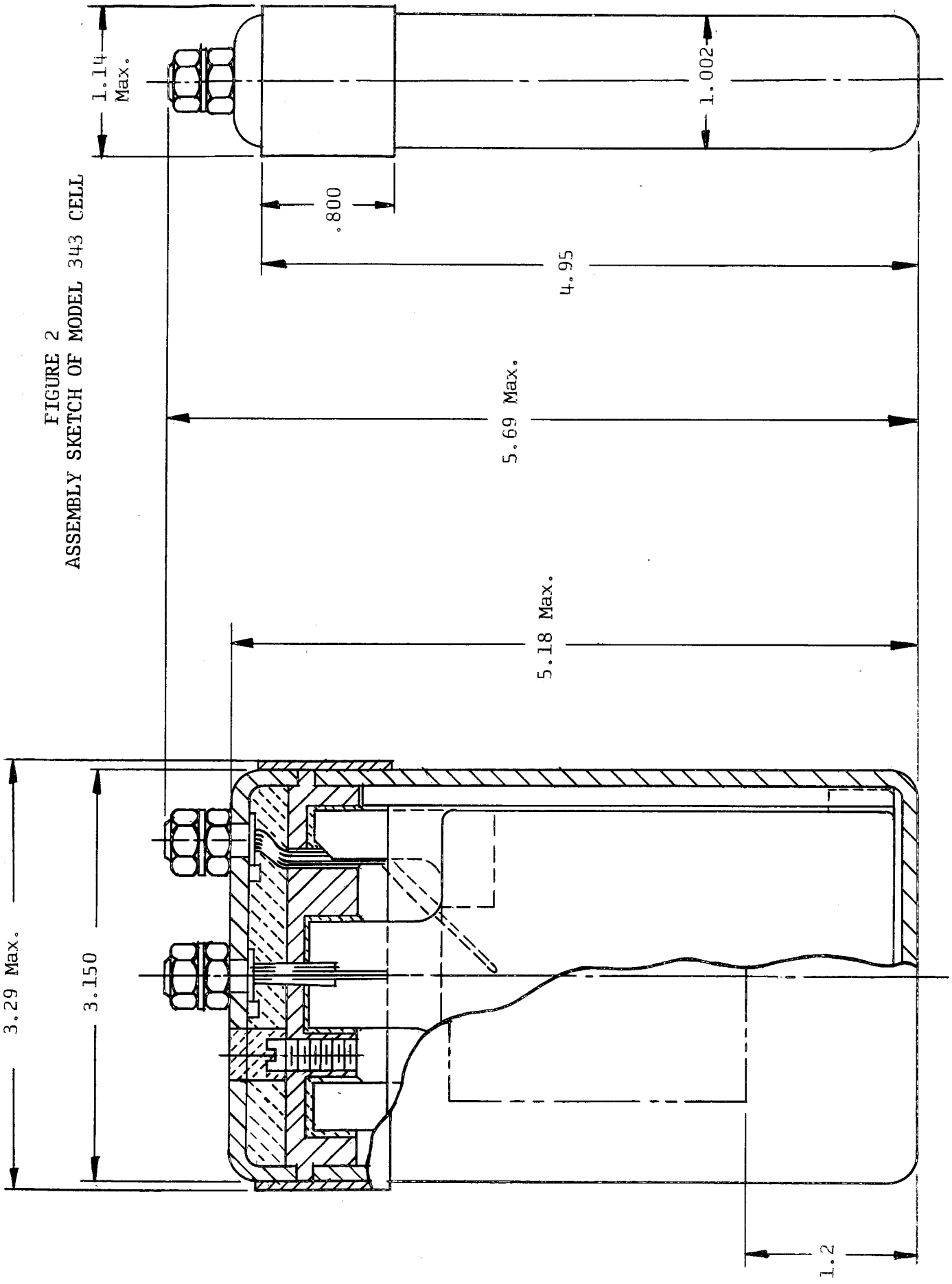


FIGURE 2
ASSEMBLY SKETCH OF MODEL 343 CELL

Four 55-mil positive plates are each supported for the high impact by an inner perforated titanium sheet and two upper titanium struts sealed with epoxy into cavities on the underside of the subcover. An independent silver lead wire system connects the positive active material to the positive cell terminal, permitting struts to break during shock without loss of capacity.

Three full and two half negative plates (79 and 53 mils thick, respectively) are each supported at three places in the cell jar; at two bottom corners and by one strut projecting from the top center of each plate into a cavity in the subcover.

The cell separator system consists of a closed "U" fold on the positive plates having (+) to (-) one layer EM-476 polypropylene absorber and five layers Southwest Research Institute GX membrane.

Titanium positive plate reinforcements can withstand in the Model 343 an allowable tensile force of 1,080 pounds per plate for an estimated 650 pound force during the 10,000 "g" shock, or a safety factor of 66%. The tensile modulus for titanium is 15×10^6 psi, approximately 40 times greater than PPO 681-111 which failed in buckling in the Model 334 cell. The allowable buckling load for each positive plate suspended inside the "U" separator fold will be 2,050 pounds with a safety factor of 3:1.

While copper, beryllium copper, and stainless steels were investigated as negative plate reinforcements, pure silver was selected for this cell design to eliminate premature shorts developing from copper oxidation during heat sterilization and later depositing on the negative plate as copper spurs, and to meet the non-magnetic requirement of one gamma at six times the average 18-cell battery dimension. On a full negative plate having a silver sheet support structure a load of 875 pounds is allowable for an estimated load at 10,000 "g" of 740 pounds.

This marginal safety factor of 18% possibly predicts a negative plate failure mode during shock. Titanium grid structures in the positive plates may also embrittle in the cell atmosphere in spite of their Ag_2O/AgO sheath, if hydrogen rich atmospheres are formed in the cell by inefficient negative electrode operation.

III. DEVELOPMENT OF 5 AH SEALED, HEAT STERILIZABLE, HIGH IMPACT SILVER-ZINC OXIDE CELLS

Design objectives for this 5 AH sealed cell - ESB Model 344 - are based on the requirements of JPL Specification GMP 50437-DSN-C for cells capable of:

- activation, seal, heat sterilization for 120 hours at 135°C

- formation charge as an 18-cell battery check out, flight to planet on float (or on full charged stand) for 8 months.

- withstanding a landing impact of 10,000 g peak for 0.1 msec. followed by 5,000 g peak square pulse of 3 msec duration and then

- delivery of 5.0 AH at rates as high as 300 watts at a minimum voltage of 1.25 V over the temperature range 10 to 55°C.

Allotted volume for a six cell group has been designated to be a rectangular block 4.00" x 3.50" x 3.75". Development covers a period to 1 December 1967 with the delivery of 200 sealed cells to JPL.

Development to this date has reached the engineering cell assembly stage. Drawings are complete and have been reviewed by ESB Quality Assurance and Reliability at the prototype release design review. Table XXX summarizes cell physical and electrochemical design characteristics. Figure 3 is an assembly sketch of the proposed cell.

The overlapping top cover, the subcover, and the cell jar are intended to perform a reliable seal function during the 10,000 "g" shock after heat sterilization and will all be molded to close tolerances in polyphenylene oxide 531-801 material. The epoxy seal between these components and around the plate struts will consist of DOW DEN438-EK85 with catalyst CMP-30 adhesive plus a sealant to be selected by tests in progress. This sealant has the function of sealing around silver and titanium sheet struts and to PPO 531-801 over the 255°F thermal shock from the low storage temperature minimum of 20°F to the heat sterilization maximum of 275°F.

Positive plate grids will consist of .008" perforated titanium sheet with 2/0 Ag expanded grid spotwelded on either side to support the active porous Ag material. Effective plate area in six positives will be 33.8 in^2. At a maximum rate of 300 watts and 12.5 amps the cell operating current density will be 0.37 amps/in^2.

Negative plate grids will consist of a sandwich of 0.010" perforated Ag sheet with 2/0 Ag expanded grid spotwelded on both sides.

The respective plate assembly weights, their estimated "g" forces, and the calculated design weak site safety factors under the tensile stresses of the 10,000 "g" shock in the plate height dimension are:

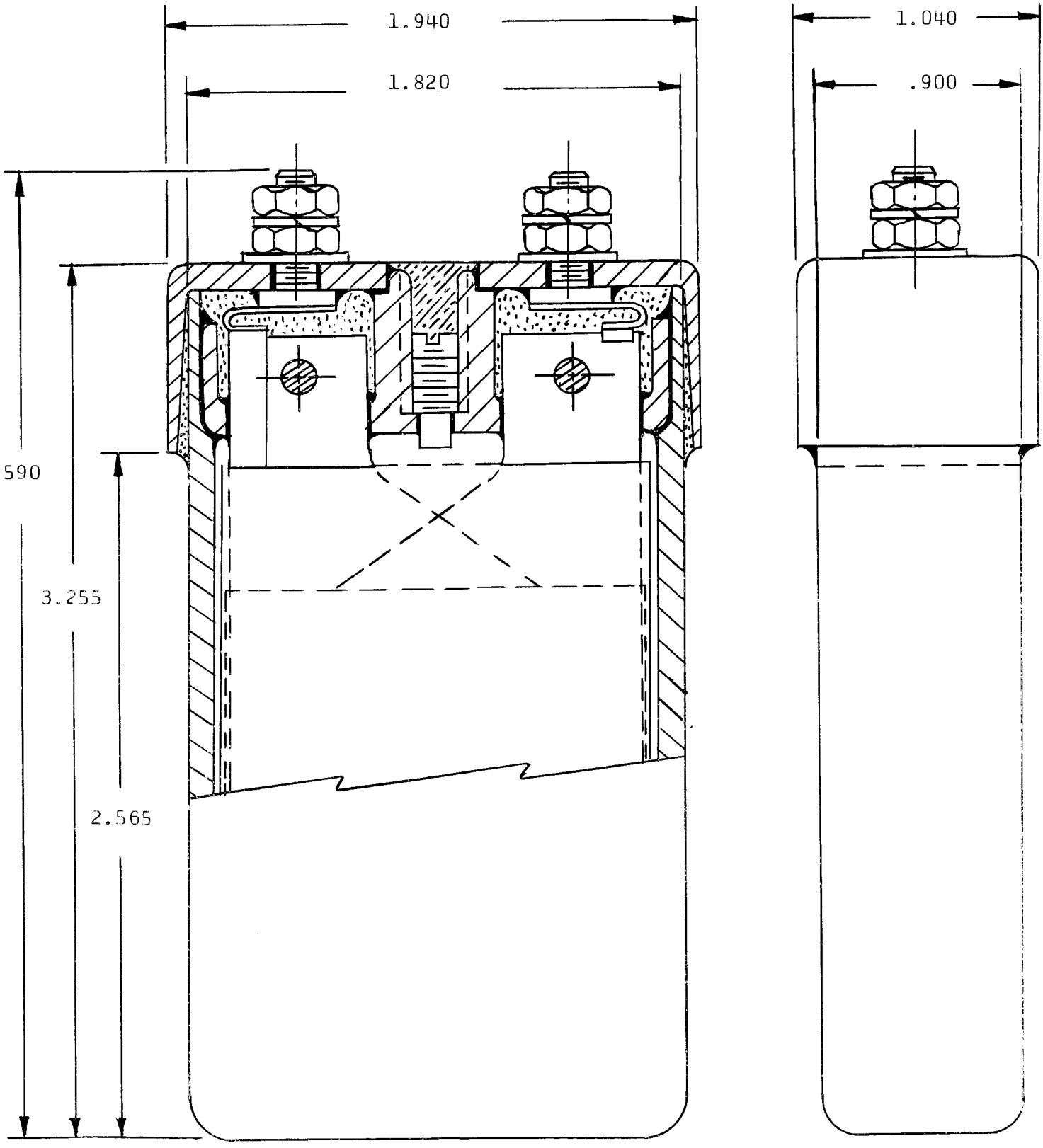
TABLE XXX

MODEL 344 5 AH HIGH RATE
HEAT STERILIZABLE, HIGH IMPACT CELL DESIGN

Cell Component	No. In Cell	Material	Dimension, Inches L	W	H	T	Weight Per Cell gms
Cell Case		PPO 531-801	.90	1.80	3.14	.12	32.1
Subcover		PPO 531-801	.78	1.68	.58	.10	8.0
Top Cover & Plug		PPO 531-801	1.04	1.94	.69	.10	7.7
Cement		Epoxy					5.0
Sealant		Epoxy					10.0
Cell Pack							
Positive Blanks	6	Porous Ag		1.50	1.88	.020	23.4
Positive Grids	6	Ti sheet/Ag grids		1.50	2.80	.031	14.1
Positive Absorber	3	EM-476 (1 L)	5.00	3.07		.003	1.1
Positive Membrane	3	SWRI-GX (4 L)	12.8	4.90		.003	4.5
Negative Retainer	7	EM-476 (1 L)	3.9	3.4		.003	1.4
Negative Active Material	7	ZnO, "X"		1.50	1.88	.023	22.1
Negative Grids	7	Ag sheet & grid		1.50	1.88	.013	47.6
Electrolyte, 27.5 cc		40% KOH					38.6
Hardware (Terminals, hex nuts, washers)	--						10.0
Cell Dimensions							
Overall			1.04	1.94	3.59		
Case Only			.90	1.82	3.26		
Volume, in^3	5.3						
Cell Weight, Wet Sealed							225.6 gms (0.5 lb max)
Capacity, Expected, AH	6.8						
Energy, Expected, WH	9.0						
Energy Density, WH/lb, WH/in^3	18.0 1.7						

FIGURE 3

ASSEMBLY SKETCH OF PROPOSED 5 AH CELL



	Positive	Negative
Plate Weight, gms	6.3	10.2
Shock Force, lbs.	138	138
Design Safety Factor, %	60	42
Weak Site	Strut at seal	Top row of perforations.

Figure 4 gives assembly sketches of the positive and negative grid structures.

The ratio of active mass to total mass in this cell design is very low because of structural requirements and accounts for the low estimated energy densities of 18 WH/lb. and 1.7 WH/in^3 for a discharge capacity of 6.8 AH at a mean voltage of 1.33 volts.

To improve cell voltages the engineering cells will have only four layers of Southwest Research Institute film GX as the semi-permeable membrane. From positive plate to negative plate the total wet thickness will thus be:

1L	EM 476 Absorber	.003
4L	SWRI-GX	.012
	Total Wet Thickness:	.015 inch

This design choice may compromise cell total life for high rate performance; however, this choice is necessary to achieve the 300 watt discharge rate at no sacrifice in impact resistance.

IV. EVALUATION OF SWRI-GX FILM

Screening tests were conducted on four samples of four lots of Southwest Research Institute separator film. The observed data is summarized in Table XXXI with the individual lot means and the calculated four lot mean. Plus or minus three standard deviation limits are given for each test parameter as tentative ESB Quality Control acceptance limits. The apparent increase in dry thickness from lot GX85 to lot GX88 is the result of wrinkling. When the film is wetted with 40% KOH, wrinkles tend to smooth out and the thickness measurement of four layers contains less wrinkle factor included in the single layer thickness estimate. The wet thickness measurements indicate that a safe design wet thickness value would be 3.1 mils per single layer. ESB Models 343 and 344 are based on 3.0 mils per single layer.

FIGURE 4

5 AH CELL PLATE SUPPORT STRUCTURES
FOR 10,000 "g" IMPACTS

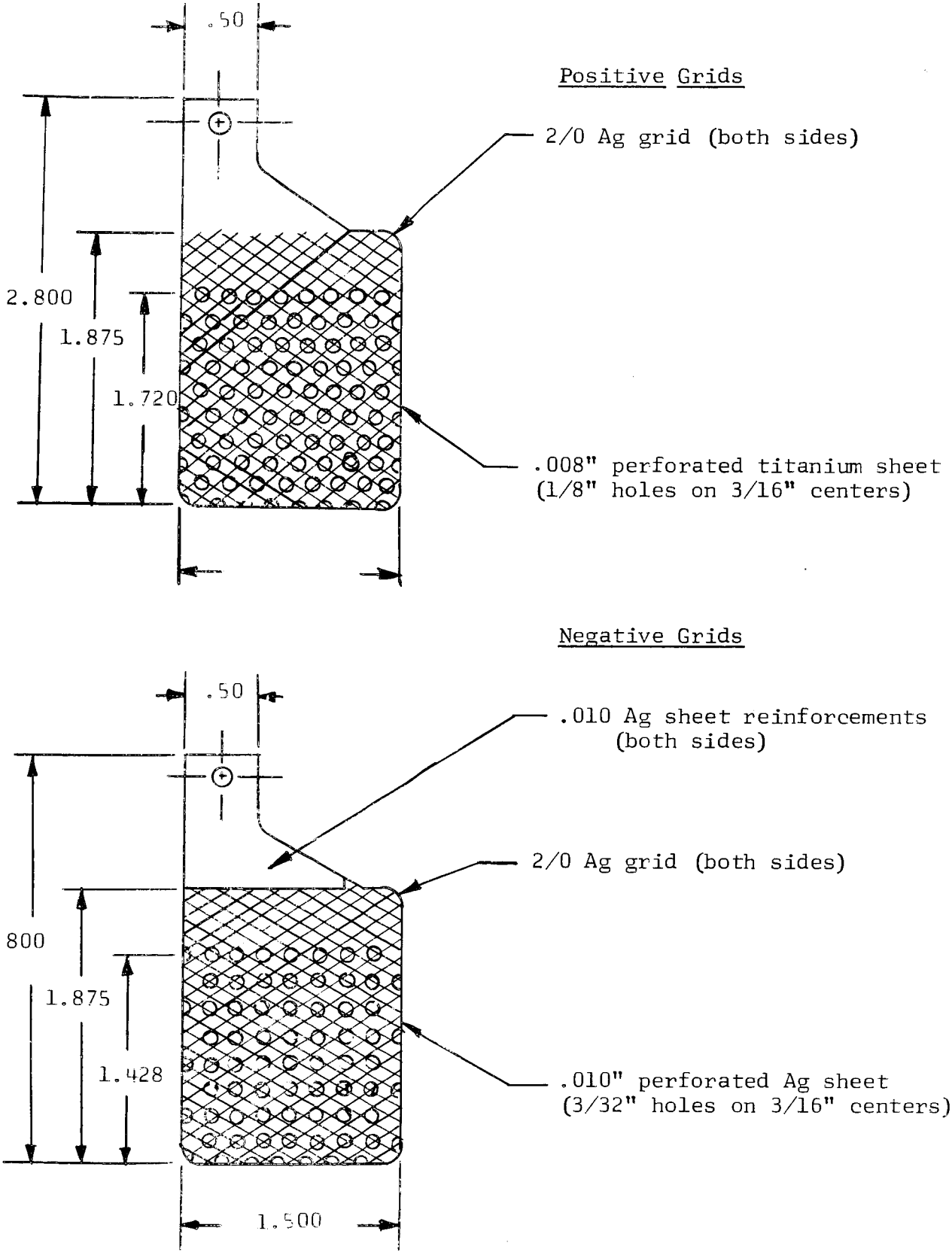


TABLE XXXI

ACCEPTANCE TEST RESULTS SWRI-GX
SEPARATOR MATERIAL*

Lot No.	Spec. No.	Dry Thickness/ mils	Wet Thickness/ mils	Length Change + % (Roll)	Width Change ± %	Thickness Change %	Weight Dry mgm/in^2	Weight Wet mgm/in^2	Porosity %	Pore Size Å	Unit Electrolyte Absorption (gms/gms)
GX 85	1	1.3	2.5	5.0	7.0	100	21	48	50.	2.0	1.5
	2	1.3	2.5	5.0	6.0	100	21	47	50.	1.8	1.5
	3	1.4	2.5	5.0	7.0	79	21	47	49	1.8	1.5
	4	1.8	2.5	5.0	7.0	43	21	48	51	1.8	1.6
	$\bar{X}$	1.4	2.5	5.0	6.8	81	21	48	50	1.8	1.5
GX 86	1	1.4	1.5	6.0	7.0	7	22	46	77	2.0	1.4
	2	1.4	2.0	4.0	6.0	43	22	45	54	1.7	1.2
	3	1.8	2.3	5.0	7.0	29	23	47	50	1.9	1.3
	4	1.3	2.0	4.0	6.0	60	22	47	59	2.0	1.4
	$\bar{X}$	1.5	1.9	4.8	6.5	35	22	46	60	1.9	1.3
GX 87	1	1.5	1.8	8.0	6.0	20	26	52	71	1.7	1.3
	2	1.5	2.0	7.0	6.0	33	24	49	60	1.5	1.3
	3	1.5	2.0	6.0	6.0	33	23	47	58	1.7	1.3
	4	1.5	2.3	5.0	5.0	53	24	48	50	1.5 1.6	1.2
	$\bar{X}$	1.5	2.0	6.5	5.8	35	24	49	60	1.6	1.3
GX 88	1	2.0	2.3	7.0	8.0	13	26	53	61	2.0	1.4
	2	2.0	2.3	6.0	7.0	13	25	53	59	2.0	1.4
	3	2.0	2.3	6.0	6.0	13	24	52	58	1.7	1.4
	4	2.0	2.5	4.0	6.0	25	23	52	60	1.2	1.5
	$\bar{X}$	2.0	2.3	4.8	7.0	16	24	53	60	1.7	1.4
All	$\bar{X}$	1.6	2.2	5.5	6.4	42	23	49	57	1.8	1.4
	ρ	.12	.30	1.2	0.7	30	1.7	2.7	7.9	0.2	0.12
± 3ρ	Max.	2.0	3.1	9.1	8.5	132	28	57	80	2.5	1.8
	Min.	1.2	1.3	1.9	4.3	0	18	41	34	1.2	1.0

(*) Tests per ESB MS-263 using 40% KOH.

CONCLUSIONS

Cell Assembly

1. Sixteen laboratory cells have been made with components regarded as most suitable for resistance to the effects of heat sterilization. The cells were sealed, sterilized, formed, cycled satisfactorily through four cycles, and are now on stand or float.

Electrodes

2. Compound 323-43 does not appear to be a cause of excessive hydrogen evolution from the negative during formation.

3. Amalgamation of the silver negative grid greatly reduces gassing of the negative electrode during formation.

4. There is evidence that elimination of Teflon from the negative mix decreases hydrogen evolution from the negative during formation.

Electrolyte

5. No change in conclusion of First Annual Report.

Separator

6. At JPL's request, SWRI-GX is being used in cells of recent construction. This material is chemically similar to RAI-116 and appears to be more uniform.

Absorber

7. EM-476 is still the recommended absorber. Other activities have prevented more thorough investigation of modified EM-476.

Case Material

8. No change in conclusions of First Annual Report.

Sealing Techniques

9. The most satisfactory cover-to-case seals have been achieved by the use of an epoxy resin adhesive on PPO 531-801.

10. Hot gas welding of PPO 541-801 no longer looks promising.

11. A new silver terminal design has given crack-free covers in PPO 541-801, but not in PPO 531-801.

Cell Design and Fabrication

12. Various components of the Model 334 cell failed mechanically at the 10,000 g peak impact. The nature of the failures suggests the need to reinforce electrodes with metal sheets rather than with plastic.

FUTURE WORK

Cell Assembly

1. A factorial experiment is being implemented to determine whether amalgamation of the silver negative grid, the presence of Teflon in the negative mix, and cell pack tightness are actually significant variables in the control of excessive pressure build-up during formation.

Electrodes

2. As indicated in the preceding paragraph, negative electrodes with amalgamated silver grids and negative mixes with and without Teflon will be under examination.

Separators

3. Continue observation of the performance of SWRI-GX in both silver-zinc and silver-cadmium cells.

Absorber

4. It is planned to resume work on modified EM-476 when manpower commitments permit.

Case Material and Sealing Technique

5. The necessity for further work on case materials is not foreseen at this time, but additional studies in sealing techniques may be required as designs are developed for the prototype cells.

Cell Design and Fabrication

6. Work will continue on constructing and testing the Model 343 cell having the metal sheet supports for electrodes and using the JPL cell case.

7. Production of 200 sealed five amp-hr cells will be started as required by a recently added task.